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IS 13402 (1992): Pesticide - Anilophos, Technical [FAD 1: Pesticides and Pesticides Residue Analysis]

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भारतीय मानक

कीटनाशक – ऐनिलोफास, तकनीकी – विशिष्टि

Indian Standard

PESTICIDE — ANILOPHOS, TECHNICAL — SPECIFICATION

UDC 632·951 ANI

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

September 1992 Price Group 2

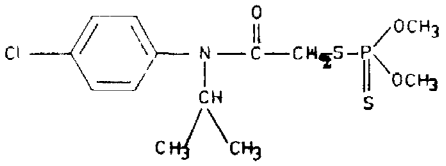
Pesticides Sectional Committee, FAD 1

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Pesticides Sectional Committee had been approved by the Food and Agriculture Division Council.

Anilophos, technical is employed in the preparation of herbicidal formulations.

Anilophos is the proposed common name by the International Organization for Standardization (ISO) (draft English-ISO) for *S* — 4 — chloro — *N* — isopropyl carbaniloylmethyl *O*, *O* — dimethyl phosphorodithioate. The empirical and structural formulae and the molecular mass are given below:

Empirical Formula	*Structural Formula*	*Molecular Mass*
$C_{13}H_{19}ClNO_3PS_2$		367·5

In the preparation of this standard, due cosideration has been given to the provisions of the *Insecticides Act, 1968* and the Rules framed thereunder. However, this standard is subjected to the restrictions imposed under these wherever applicable.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed, or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

PESTICIDE — ANILOPHOS, TECHNICAL — SPECIFICATION

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for anilophos, technical.

2 REFERENCES

The Indian Standards listed below are necessary adjuncts to this standard:

IS No.	*Title*
1070 : 1992	Reagent grade water — Specification (*third revision*)
6940 : 1982	Methods of test for pesticides and their formulations (*first revision*)
8190 (Part 1) : 1988	Requirements for packing of pesticides: Part 2 Solid pesticides (*second revision*)
10946 : 1984	Methods of sampling for technical grade pesticides.

3 REQUIREMENTS

3.1 Description

The material shall be in the form of a yellowish to brown semi-solid substance. Some liquid fraction may also be present in the container, particularly in hot weather. It shall be free from extraneous impurities or added modifying agent(s).

3.2 The material shall also comply with the requirements given in Table 1.

4 PACKING

The material shall be packed in mild steel drums provided with a LDPE liner of thickness not less than 0·062 mm. The general requirements as given in 2 of IS 8190 (Part 1) : 1988 shall also be followed.

5 MARKING

The container shall bear legibly and indelibly the following information in addition to any other information as is required under the *Insecticides Act, 1968* and Rules framed thereunder:

a) Name of the material;

b) Indication of the source of manufacture;

c) Date of manufacture;

d) Date of expiry;

e) Batch number;

f) Net mass of the contents;

g) Anilophos content, percent (*m/m*); and

h) The minimum cautionary notice as worded under the *Insecticides Act, 1968* and Rules.

6 SAMPLING

Representative samples of the material shall be drawn as prescribed in IS 10946 : 1984.

7 TESTS

7.1 Tests shall be carried out by the methods referred to in col 4 and 5 of Table 1.

7.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070 : 1992) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

Table 1 Requirements for Anilophos, Technical

(*Clauses* 3.2 *and* 7.1)

Sl No.	Characteristic	Requirement	Method of Test, Ref to	
			Annex of this Standard	Cl No. of IS 6940 : 1982
(1)	(2)	(3)	(4)	(5)
i)	Anilophos content, percent by mass, *Min*	93·0	A	—
ii)	Material insoluble in acetone, percent by mass, *Max*	0·5	—	9
iii)	Moisture content, percent by mass, *Max*	0·5	—	4.1
iv)	Acidity (as H_2SO_4), percent by mass, *Max*	0·3	—	11.3

ANNEX A

[*Clause* 2.2, *Table* 1, *Item* (i)]

DETERMINATION OF ANILOPHOS CONTENT

A-1 GENERAL

Anilophos content may be determined by either of the two methods, namely, high performance liquid chromatography (*see* A-2) or gas liquid chromatography (*see* A-3).

A-2 HIGH PERFORMANCE LIQUID CHROMATOGRAPHY

A-2.1 Principle

A high performance liquid chromatograph (HPLC) unit with an ultra-violet (UV) detector is used for the determination. Using solutions containing known amounts of anilophos reference standard and internal standard, the response factor (*Rf*) for anilophos in the internal standard is arrived at. A solution containing known mass of the anilophos sa mple and internal standard is injected subsequently. The percentage of anilophos in the sample is then computed by the standard relationship.

A-2.2 Apparatus

A-2.2.1 *HPLC Unit*

Equipped with a UV detector and a printer-plotter-*cum*-integrator. The HPLC operating conditions as suggested below can be changed in the equipment employed, provided standardization is done:

Column : Stainless steel, 25 cm length and 4·6 mm ID; packed with silica 5 μm particle size.
Detector : Ultra-violet (at 254 nm)
Solvent system : Chloroform: isopropyl alcohol: iso-octane: 3:0·5: 96·5 (percent, *v/v*)
Sovent flow rate : 1·5 ml/minut
Sample size : 20 μl

A-2.3 Reagents

A-2.3.1 *Solvent System*

Mix 3 percent chloroform, 0·5 percent isopropyl alcohol and 96·5 percent iso-octane.

A-2.3.2 *Anilophos Reference Standard* of known purity.

A-2.3.3 *Internal Standard* — thymol, AR grade.

A-2.3.4 *Chloroform* — spectroscopic grade.

A-2.3.5 *Isopropyl Alcohol* — spectroscopic grade.

A-2.3.6 *Iso-octane* — spectroscopic grade.

A-2.4 Procedure

A-2.4.1 *Preparation of Internal Standard Solution*

Weigh accurately 250 mg of thymol into a 50-ml volumetric flask containing small quantity of solvent system, mix well and make up the volume with solvent system.

A-2.4.2 *Preparation of Reference Standard Solution*

Weigh accurately 2 g of anilophos reference standard into a 100-ml volumetric flask, dissolve in toluene, shake well and make up the volume with toluene. Take out 10 ml of the solution with a pipette into a 50-ml volumetric flask and make up the volume to the mark with the solvent system. Again take out 10 ml of this solution into another 50-ml volumetric flask, add 10 ml internal standard solution, make up the volume to the mark with the solvent system.

A-2.4.3 *Preparation of Sample Solution*

Warm up about 100-150 g anilophos, technical sample in a container, on a water-bath maintained at 50°C and stir thoroughly with a glass-rod for a few minutes to melt the mass into a homogeneous state. Transfer about 2 g of the molten mass into a beaker with a previously weighed glass-rod. Allow the beaker and the contents to cool at room temperature and weigh till a constant weight is obtained. Dissolve the sample in toluene and quantitatively transfer with care into a 100-ml volumetric flask. Wash the beaker several times with toluene, adding the washings to the flask. Make up the volume with toluene and shake well. Take out 10 ml of the solution with a pipette into a 50-ml volumetric flask and make up the volume to the mark with the solvent (*see* A-2.3.1). Mix well. Again take out 10 ml of this solution into another 50-ml volumetric flask, add 10 ml internal standard solution and make up the volume to the mark with the solvent system.

A-2.4.4 *Estimation*

A-2.4.4.1 Introduce 20 μl anilophos reference standard solution into the HPLC unit, and from the chromatogram, calculate the response factor (Rf_1) as follows. Repeat the injection till Rf_1 values agree well within ±2 percent.

$$\text{Response factor } (Rf_1) = \frac{A_1}{A_2}$$

where

A_1 = peak area for the anilophos reference standard solution injected, and
A_2 = peak area for the internal standard solution injected.

A-2.4.4.2 Introduce 20 μl sample solution into the HPLC unit and calculate the response factor (Rf_2) as described in **A-2.4.4.1**.

$$\text{Response factor } (Rf_2) = \frac{A_3}{A_4}$$

where

A_3 = peak area for the anilophos sample solution injected, and

A_4 = peak area for the internal standard solution injected.

A-2.5 Calculation

$$\text{Anilophos content, percent by mass} = \frac{Rf_2 \times M_1}{Rf_1 \times M_2} \times P$$

where

Rf_2 = response factor for the anilophos sample;

M_1 = mass, in g, of anilophos reference standard;

Rf_1 = response factor for anilophos reference standard;

M_2 = mass, in g, of anilophos sample; and

P = percent purity of anilophos reference standard.

A-3 GAS LIQUID CHROMATOGRAPHY

A-3.1 Apparatus

A-3.1.1 *Microlitre Syringe* — 5-10 μl capacity.

A-3.1.2 *Gas Liquid Chromatograph* (*GLC*)

Equipped with a flame ionization detector (FID) and may be coupled to a printer-plotter-*cum*-integrator. The operative parameters are suggested below, which can be changed, in any other GLC equipment employed, provided standardization is done:

Column — Glass, 100 cm length and 4 mm ID packed with 3 percent SE-30 on Chromosorb WHP (100-120 mesh)

Temperatures

Column oven — 200°C
Injection port — 200°C
Detector — 200°C

Gases and Corresponding Flow Rates

Nitrogen (carrier) — 40 ml per minute
Hydrogen — 40 ml per minute
Air — 300 ml per minute

NOTE — Anilophos has been observed to decompose beyond 200°C, and therefore, it is necessary to maintain the suggested temperatures.

A-3.2 Reagents

A-3.2.1 *Toluene* — AR grade

A-3.2.2 *Internal Standard Solution* — Benzyl butyl phthalate, AR grade

A-3.2.3 *Anilophos Reference Standard* — of known purity

A-3.3 Procedure

A-3.3.1 *Preparation of Internal Standard Solution*

Weigh accurately 400 mg benzyl butyl phthalate into a 100-ml volumetric flask, dissolve in toluene and make up the volume with toluene.

A-3.3.2 *Preparation of Reference Standard Solution*

Weigh accurately 2 g of anilophos reference standard into a 100-ml volumetric flask, dissolve in toluene, shake well and make up the volume with toluene. Take out 5·0 ml of the solution with a pipette into a 25-ml volumetric flask, add 10 ml internal standard solution, make up the volume to the mark with toluene. Mix well.

A-3.3.3 *Preparation of Sample Solution*

Warm up about 100-150 g anilophos, technical sample in a container, on a water-bath maintain at 500°C and stir thoroughly with a glass-rod for a few minutes to melt the mass into a homogeneous state. Transfer accurately weighed 2 g of the molten mass into a beaker with a previously weighed glass-rod. Allow the beaker and the contents to cool to room temperature and weigh till constant weight is obtained. Dissolve the material in toluene and quantitatively transfer with care into a 100-ml volumetric flask. Make up the volume and mix well. Take out 10 ml of this solution into a 50-ml volumetric flask, add 10 ml internal standard solution, make up the volume with toluene and mix well. The final concentration of anilophos in standard and sample solutions will be around 4 mg/ml.

A-3.3.4 *Estimation*

A-3.3.4.1 Introduce 1 μl anilophos reference standard solution into the GLC unit, and from the chromatogram, calculate the response factor (Rf_1) as follows. Repeat the injection till Rf_1 values agree well within ±2 percent:

$$\text{Response factor } (Rf_1) = \frac{A_1}{A_2}$$

where

A_1 = peak area for the anilpohs reference standard solution injected, and

A_2 = peak area for the internal standard solution injected.

A-3.3.4.2 Introduce 1 µl sample solution into the GLC unit and calculate the response factor (Rf_2) as described in **A-3.3.4.1**

$$\text{Response factor } (Rf_2) = \frac{A_3}{A_4}$$

where

A_3 = peak area for the anilophos sample solution injected, and

A_4 = peak area for the internal standard solution injected.

A-3.4 Calculation

Anilophos content,

$$\text{percent by mass} = \frac{Rf_2 \times M_1}{Rf_1 \times M_2} \times P$$

where

Rf_2 = response factor for the anilophos sample;

M_1 = mass, in g, of anilophos reference standard;

Rf_1 = response, factor for anilophos reference standard;

M_2 = mass, in g, of anilophos sample; and

P = percent purity of anilophos reference standard.

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